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Submicron processing of InAs based quantum wells: A new, highly selective wet etchant for AlSb

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We describe a processing technology for patterning InAs/AlSb heterostructures far in the submicron regime. The processing is based on a new, highly selective wet etchant for AlSb. We discuss the electrical characterization of narrow ballistic channels (down to ≈140 nm width) realized with present technology, and demonstrate that the processing preserves the high mobility of the material. © *1997 American Institute of Physics.* [S0003-6951(97)03111-2]

Due to their unique properties, systems based on the two-dimensional electron gas (2DEG) present in Al(Ga)Sb/InAs heterostructures have been employed both in applications and in the investigation of fundamental electronic phenomena of very different nature. A number of field effect transistors have been realized, which can be operated at room temperature with potentially advantageous performances.[1] The pinning of the Fermi energy in the conduction band of an exposed InAs layer makes these materials ideal in the study of mesoscopic superconductivity in low dimensionality systems.[2] The presence of strong spin orbit interaction of the Rashba type[3] has led to the suggestion to use InAs heterostructures to study the influence of Berry's phase on transport phenomena.[4] The coexistence of electrons and holes in InAs/GaSb quantum wells gives an experimental handle to investigate the possibility of excitonic ground states in semimetallic systems.[5]

However, technological difficulties often make working with these materials problematic. In particular, in the experimental investigation of many aspects of the fundamental phenomena just mentioned, the ability to process Al(Ga)Sb/InAs quantum wells on a small dimension scale ($\ll 1$ μm) presents a severe limiting factor.

The simplest technique to realize samples of small dimensions is wet chemical etching.[6] It is then important to have access to a family of selective wet etchants for InAs, GaSb, and AlSb, with controllable and reproducible properties. Fairly selective solutions that etch InAs in a controllable way are available.[7] However, all selective wet etchants for AlSb and GaSb known to us are affected by serious problems which impede their applications in the submicron range (see Ref. 8 for specific details).

In this letter we describe a recently developed technological process that makes it possible to shape InAs/AlSb quantum wells far into the submicron range. The process relies on a new, very selective wet etching technique for AlSb. This technique is based on the use of a solution of strongly water diluted HF (hydrogen fluoride), further diluted in ethanol. We have studied the action of this solution on several different AlSb/InAs heterostructures and we have found that it is very reproducible. In order to demonstrate that the present technology does not spoil the properties of the material, we also present results on the electrical characterization of narrow (<150 nm) ballistic channels. The technology described here has been already used successfully to realize superconducting interferometers operating in the ballistic regime[9] and small Aharanov–Bohm rings.[10]

The materials employed in the present investigations consist of a GaSb cap layer (3 nm), covering a 50 nm AlSb layer, which provides the confinement for the electrons hosted in the next 15 nm InAs layer. Underneath the InAs another AlSb layer is present. The heterostructures were grown on a GaAs substrate.

We have used electron beam lithography to define etching masks in a conventional electron beam resist (PMMA). After exposure and development (developer IPA:MIBK 1:1) of the mask pattern, samples are loaded into a rf reactive ion etcher and exposed to a low voltage (dc self-bias voltage ≈60 V, power 20 W) oxygen plasma for ≈25 s (O_2 pressure ≈10 mbar). The plasma removes thin spots of PMMA still present on the surface after development, that would otherwise make the etching inhomogeneous.

The samples are subsequently loaded into a second vacuum chamber where the 3 nm GaSb cap layer is removed mechanically in the regions exposed through the PMMA mask, by ion bombardment in a rf generated Ar plasma (dc self bias ≈400 V; power ≈400 W; Ar pressure ≈25 μbar). At an early stage of this work we have also tried to do this operation with a Kaufmann source and we have seen that, contrary to the Ar plasma, this introduces a high degree of inhomogeneity in the subsequent processing steps.

The following step consists of the AlSb layer etching. It is known[8] that a highly water diluted solution of HF containing H_2O_2 etches AlSb and it is moderately selective with respect to InAs. With decreasing the concentration of H_2O_2 the etching speed of this solution decreases and its selectivity increases. We have tested a HF:H_2O 1:700 solution (with no H_2O_2) and we have observed that, even though it actually etches AlSb rather selectively with respect to InAs, its action is highly irreproducible, very sensitive to defects and material dependent. Furthermore, since water diluted HF etches AlSb extremely fast, it is impossible to control in the submicron domain.

We have found that a substantial improvement of the

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etching properties of water diluted HF is obtained if this solution is further diluted in ethanol. Specifically, we have found that the solution obtained by diluting one part of HF:H_2O 1:700 in ten parts ethanol [(HF:H_2O 1:700):ethanol 1:10; PH≈3] is an excellent etchant for AlSb. It has several attractive properties: (1) selectivity [this solution etches AlSb 100 times (or more) faster than InAs and GaSb]; (2) small underetching (the underetching is comparable to the etched depth); (3) material independent and reproducible (it has been tested on four different wafers with identical reproducible results); (4) high quality edges (the edges of the etched structures are straight, and their profile vertical).

We have observed that the etching speed of the solution is influenced by the presence of PMMA. In particular, the etching proceeds more rapidly within a ≈5 μm distance from the PMMA. Most of our work has been done on small structures in which every part of the exposed AlSb was within this distance from a large region covered with PMMA. In this case the etching speed is ~0.5 nm/s. We have also tested the etching in much larger areas (≈100×100 μm^2) and we have seen that, in that case, a solution containing a larger amount of HF:H_2O [(HF:H_2O 1:700):ethanol 1:5] gives the best homogeneous results, with approximately the same speed. The difference in etching speed close and far away from PMMA can cause some problems if one wants to etch, at the same time, large areas and small features. In that case it is recommendable to separate the etching in two distinct steps.

During the etching, the AlSb surface looks very rough if inspected with an optical microscope. The color is dark reddish. The etching stops at the InAs layer. The exposed InAs surface is smooth and its color is brighter than that of the AlSb. This difference in morphology and color makes it easy to determine when all the AlSb has been completely removed.

The final step necessary to define the structure is the wet etching of the InAs layer, done using a known succinic acid based solution.[11] The selectivity of this solution with respect to GaSb and AlSb is good, but not exceptional, so that the AlSb edges are also etched during this processing step. This is the limiting factor of the spatial resolution of the described technology. Presently, the smallest structures that we have been able to realize reproducibly have a width of ≃130 nm.

A remark is necessary on the only problematic aspect related to the use of the (HF:H_2O):ethanol solution. We have observed that a small amount of water dissolved in ethanol acts as a developer for PMMA.[12] As a consequence, the PMMA mask can be severely damaged during the etching of the AlSb layer, especially in those regions that have received a considerable amount of background exposure due to proximity effect during electron beam writing. This effect is irrelevant when only etching steps are involved. However, if the etching of the AlSb layer is followed by metallization and a subsequent liftoff step, problems can arise due to the poor edge profile of the PMMA. In order to minimize these problems we have used a thick (≈350 nm) PMMA mask. In this way it is possible to achieve proper liftoff on a scale of ≈0.3 μm.

In concluding the description of the etching process it is important to stress the following. AlSb is a rather unstable material that easily oxidizes when exposed to air. Especially when working on structures of very small dimensions, it is extremely important to protect the sides of the structures to prevent a severe deterioration of the sample. We have found that covering our samples with PMMA (baked at 180 °C for ≈45 min) immediately after the end of the etching process provides a very effective means of protection (no sign of degradation 4 months after samples preparation).

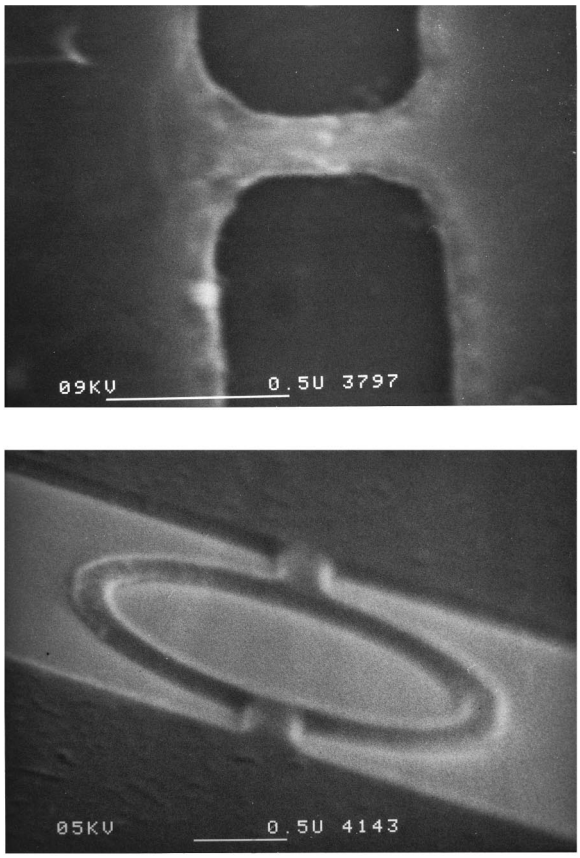

FIG. 1. One of the channels used in the electrical characterization (top) and an Aharanov–Bohm ring (bottom) realized with the present technology. In both pictures the white bar corresponds to 0.5 μm.

The electrical characterization of the technology discussed above has been done on channels (Fig. 1) etched in a high quality two-dimensional electron gas. The electron density of the material, $N=1.75\times10^{16}$ m^{-2}, and the mean free path, $l_e=1.9$ μm, have been obtained from measurements on a large Hall bar. Since the length of the channel is ≈0.5 μm, much shorter than the mean free path, we expect transport to occur in the ballistic regime. This has been demonstrated by magnetoresistance measurements.

Figure 2 shows the magnetoresistance of channels of different width (550, 360, and 140 nm). Measurements have been performed at 4.2 K with a standard lock-in technique, in a four probe configuration. In all the measurements a series resistance of 120–150 Ω is present at $B=0$, due to the wide 2DEG region in between the channels and the probes. We note that the measured resistances scale nicely with the width of the channel, providing a first indication of the absence of diffusive scattering at the samples edges. A negative magnetoresistance at low field, followed by a crossover to positive magnetoresistance is observed in all samples. This

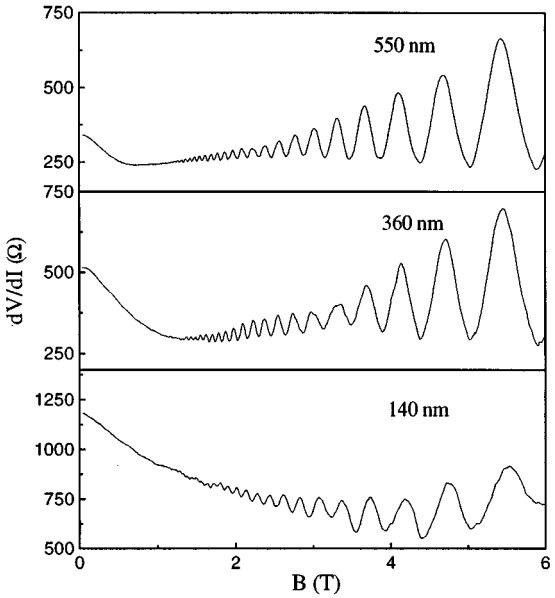

FIG. 2. Magnetoresistance of three channels of different width: 550, 360, and 140 nm.

behavior is the one expected for ballistic channels measured in four probe longitudinal configuration (i.e., the path of the injected current and of the measured voltage do not cross), as follows from the analysis of van Houten *et al.*[13] The crossover occurs at the value of magnetic field for which the electron cyclotron diameter equals the width of the channel. That is why for larger channels the crossover occurs at a smaller field.[14]

Measurements in a Hall configuration (i.e., the path of the injected current and of the measured voltage cross each other) show that the electron density in the channels is slightly lower that in the unprocessed regions (-20% in the 140 nm, -8% in the 360 nm, and -5% in the 550 nm). This is consistent with the observed positive slope observed at high field in the measurements shown in Fig. 2.[15] The Shubnikov–de Haas oscillations observed at high field (Fig. 2) are due to the series resistance. In fact the electron density obtained from their period in $1/B$ is exactly 1.75×10^{16} m^{-2}, the same measured in the large Hall bar.

The measured resistance at zero field agrees within better than 10% with the calculated Sharvin resistance in all the measured samples. This observation proves that scattering from the edges of the channels is predominantly specular and is consistent with the observed low field behavior: narrow ballistic channels with diffusive scattering from the edges should show a positive magnetoresistance at low field,[16] that is clearly not present in our data.

The ballistic nature of transport in very small samples has also been demonstrated by the measurements on superconducting interferometers,[9] made with the same high quality material discussed here. In that case point contacts of very short length have been realized and, probably because of this, magnetic depopulation of subbands has also been observed.

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